



May 2, 2014

Mr. Dwight Leisle
Port of Portland
7200 NE Airport Way
Portland, Oregon 97218

Re: Proposed Surface Soil Sampling — Operable Unit 5
Swan Island Upland Facility
Portland, Oregon
ECSI No. 271
1056-03

Dear Mr. Leisle:

This letter presents the scope for proposed surface soil sampling activities to support the preparation of a revised Source Control Measure Work Plan (the Work Plan) for Operable Unit 5 (the Facility or OU5) at the Swan Island Upland Facility (SIUF) in Portland, Oregon (Figures 1 and 2). The Port of Portland (Port) is under a Voluntary Cleanup Program (VCP) Agreement with the Oregon Department of Environmental Quality (DEQ) for Remedial Investigation (RI), Source Control Measures (SCMs), and Feasibility Study (FS) at the Facility (dated July 24, 2006). The proposed activities presented in this letter include collection of surface soil samples for chemical analysis.

BACKGROUND

A Source Control Measure (SCM) evaluation (Ash Creek, 2012) was completed to identify an appropriate SCM for the OU2 riverbank (now OU5). The objective of the SCM is to prevent erosion of soil in the vicinity of RB-9 and RB-10 (Figure 3). The recommended SCM for OU5 soils was riprap armoring and re-grading/re-vegetation in the vicinity of RB-9 and RB-10. This alternative was selected because it provides a low-cost, long-term erosion control solution; it is highly implementable; and it is compatible with existing conditions and potential in-water remediation. The Port prepared a Work Plan for this proposed alternative (Apex, 2014). Following is a bulleted summary of the comments provided by DEQ in a letter dated March 27, 2014.

- The Work Plan does not address areas outside of Erosion Scarp L and Erosion Scarp M (see SCM locations proposed on Figure 3).
- The focused sampling completed on the riverbank and upland area suggests that the riverbank below the Daimler Trucks North America Leasehold could be impacted by the upland activities. Elevated levels of metals (arsenic, copper, lead and zinc), polycyclic aromatic hydrocarbons (PAHs), and polychlorinated biphenyls (PCBs) have been observed in several samples collected from this area.
- DEQ expected that the Work Plan would include SCMs to address areas outside of these Erosion Scarps L and M, and would also include a long-term bank stability monitoring program or that the Port would propose a Work Plan to address this data gap.

The activities presented in this letter are proposed to address the data gap identified by DEQ.

PROPOSED SAMPLING ACTIVITIES

Preparatory Activities

The following activities and schedule coordination will be completed in preparation for the field work.

- **Health and Safety Plan (HASP).** Apex Companies, LLC (Apex) will update the HASP for its personnel involved with the project.
- **Coordination of Facility Access.** The work activities will be conducted in coordination with the Port.
- **Vegetation Removal.** Vegetation will be removed to provide access to the sampling area. The removal will focus on invasive species and grasses. Trees and shrubs will not be removed.

Surface Soil Sampling

The following protocol was prepared based on the *ITRC Technical and Regulatory Guidance Incremental Sampling Methodology* (dated February 2012). Surface soil samples will be collected from one decision unit using an incremental sampling technique to assess the extent of contaminants of potential concern (Figure 3). The sampling will be completed between the fence line and the approximate Ordinary Line of High Water (OLHW).

Soil samples will be collected using the following protocols.

- The sample locations will be established using a high-accuracy, handheld global positioning system (GPS) device (Trimble® GeoXH™). Hand taping methods will be employed to augment the use of the GPS in areas of reduced satellite coverage.
- The incremental sample will consist of 30 soil increments collected from a randomly selected quadrant within the grids presented on Figure 3. If the proposed sample location is not sampleable (e.g., due to the presence of armor rock, significant woody debris, etc.), the sample location will be moved to a different randomly selected quadrant within the grid, as necessary. Final sample locations will be documented in field notes.
- The samples will be collected from the top 6 inches of surface soil after removing vegetation. The samples will be collected with a 0.5-inch-diameter cylindrical stainless steel sampler. Multiple aliquots will be collected at each location (within a 5-foot diameter) in order to collect sufficient volume for analysis.
- Non-disposable items (e.g., sampler, spoons, bowls, etc.) will be cleaned by washing in a detergent (Alconox®) solution, rinsing with tap water, followed with a deionized water rinse prior to initiating sampling and between sampling locations.
- Given the nature of the contaminants of concern under evaluation, field screening will not be conducted.
- At each location, approximately 450 grams of soil will be collected into a stainless steel bowl and thoroughly homogenized. Approximately 50 grams will be placed into the ISM sampling container. The remaining mass will be retained as a discrete sample and placed in laboratory-supplied jars (for potential follow-up analyses based on the results of ISM sample results). ISM triplicate analyses are not planned.

CHEMICAL ANALYSES

The ISM soil sample will be submitted to Apex Labs of Tigard, Oregon for chemical analyses on a normal turnaround basis for the following:

- Arsenic, Copper, Lead, and Zinc by EPA 6000,
- PCBs by EPA Method 8082, and
- PAHs by EPA Method 8270-SIM.

The requested method reporting limits (MRLs) will be consistent with the historical laboratory analyses and the concentrations will be presented to the method detection limit (MDL). The target reporting limits are presented in Table 1. The Apex Labs standard operating procedure (SOP) for incremental sample processing is included in Attachment A. Following is a bulleted approach for the ISM sample processing.

- Air drying of ISM sample at room temperature on baking sheet(s);
- Sieve sample to less than 2 millimeters;
- Spread ISM sample evenly on baking sheet covered with Teflon sheeting for two dimensional slabcake;
- Take 40 (approximately 2-gram) random increments from the ISM sample;
- Place 80 grams of material in tungsten ring and puck mill grinder and grind to fine (approximately 50- to 60-micron) powder; and
- Digest the required mass of powdered sample for each laboratory analytical method.

Laboratory quality assurance/quality control (QA/QC) will include a method blank and a batch laboratory control sample (LCS)/laboratory control sample duplicate (LCSD). One laboratory process replicate will be collected from the two dimensional slabcake prepared by the laboratory.

The laboratory analytical results for the ISM sample will be discussed with DEQ before follow-up laboratory analyses are requested.

REPORTING

The results of the sampling proposed in this letter will be presented in a data report and used to revise the Source Control Measure Work Plan for OU5.

If you have any questions regarding these activities, please contact the undersigned at (503) 924-4704.

Sincerely,



EXP 12/31/2014

Michael J. Pickering, R.G.
Senior Associate Hydrogeologist

REFERENCES

- Apex, 2014. Source Control Measure Work Plan, Swan Island Upland Facility. January 17, 2014.
- Ash Creek, 2012. Source Control Alternatives Evaluation, Operable Unit 2, Swan Island Upland Facility, Portland, Oregon. November 16, 2012.
- DEQ/EPA, 2005. Portland Harbor Joint Source Control Strategy – Final (Table 3-1 Updated July 16, 2007). December 2005.
- DEQ, 2014. DEQ Review “Source Control Measure Work Plan Swan Island Upland Facility Operable Unit 5” ECSI No. 271. March 27, 2014.

ATTACHMENTS

Table 1 – Target Reporting Limits

Figure 1 – Facility Location Map

Figure 2 – Facility Vicinity Plan

Figure 3 – Proposed Sampling Plan

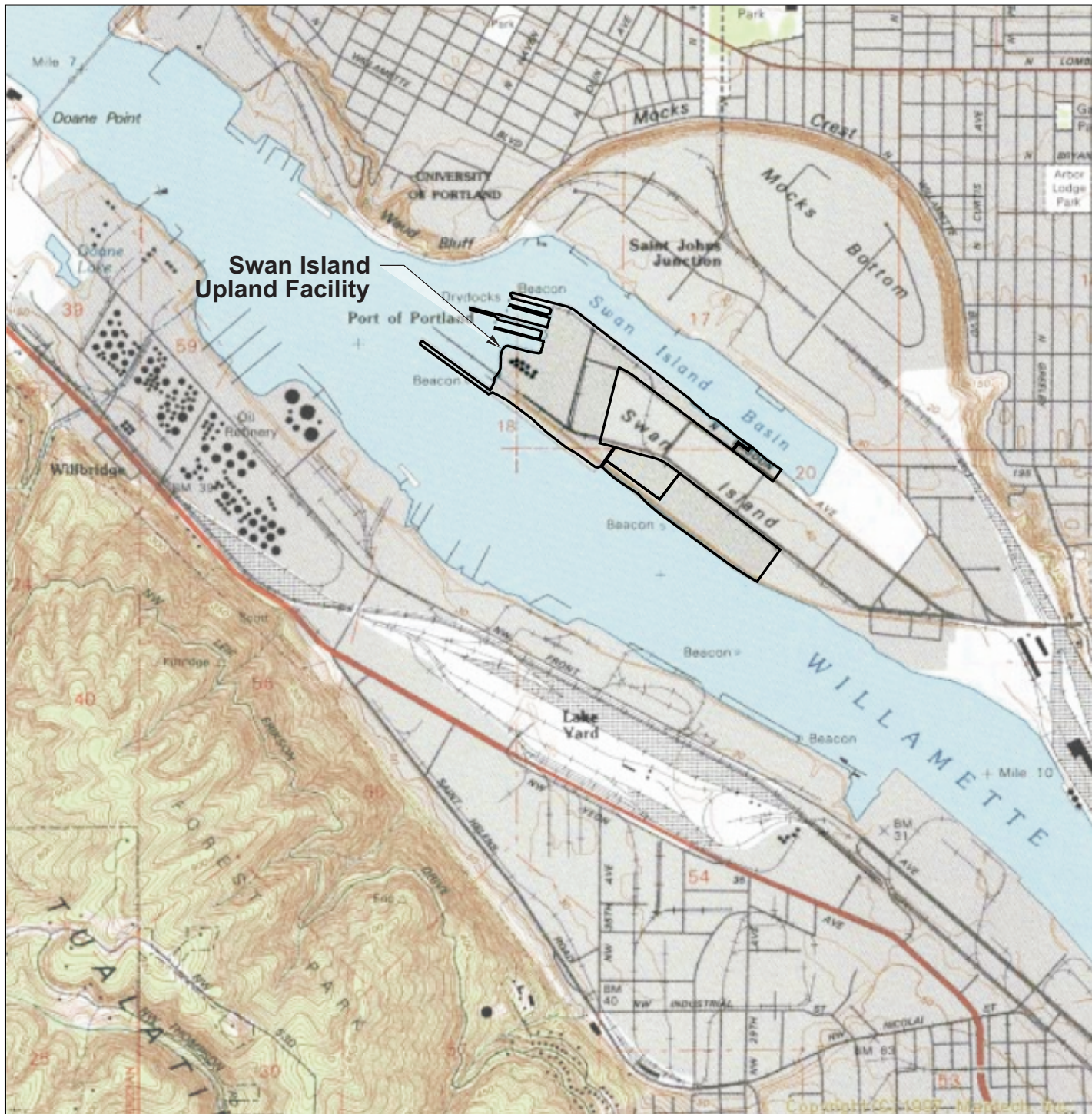
Attachment A – Apex Labs Standard Operating Procedure – Incremental Sampling Methodology

Table 1 - Target Reporting Limits
Swan Island Upland Facility, OU5
Portland, Oregon

Analyte	Soil			
	Units	MDL	MRL	JSCS
<i>Metal (EPA 6020)</i>				
Arsenic	mg/kg	0.25	1.0	7
Copper	mg/kg	0.5	1.0	149
Lead	mg/kg	0.1	0.2	17
Zinc	mg/kg	2.0	4.0	459
<i>Polycyclic Aromatic Hydrocarbons (EPA Method 8270-SIM)</i>				
1-Methylnaphthalene	ug/kg	5.0	10	--
2-Methylnaphthalene	ug/kg	5.0	10	200
Acenaphthene	ug/kg	5.0	10	300
Acenaphthylene	ug/kg	5.0	10	200
Anthracene	ug/kg	5.0	10	845
Benzo(a)anthracene	ug/kg	5.0	10	--
Benzo(a)pyrene	ug/kg	5.0	10	--
Benzo(b)fluoranthene	ug/kg	5.0	10	--
Benzo(ghi)perylene	ug/kg	5.0	10	--
Benzo(k)fluoranthene	ug/kg	5.0	10	--
Benzo(b+k)fluoranthene(s)	ug/kg	10.0	20	--
Chrysene	ug/kg	5.0	10	1,290
Dibenz(a,h)anthracene	ug/kg	5.0	10	--
Fluoranthene	ug/kg	5.0	10	2,230
Fluorene	ug/kg	5.0	10	536
Indeno(1,2,3-cd)pyrene	ug/kg	5.0	10	--
Naphthalene	ug/kg	5.0	10	561
Phenanthrene	ug/kg	5.0	10	1,170
Pyrene	ug/kg	5.0	10	1,520
<i>Polychlorinated Biphenyls (EPA Method 8082)</i>				
Aroclor 1016	ug/kg	5.0	10	530
Aroclor 1221	ug/kg	5.0	10	--
Aroclor 1232	ug/kg	5.0	10	--
Aroclor 1242	ug/kg	5.0	10	--
Aroclor 1248	ug/kg	5.0	10	1,500
Aroclor 1254	ug/kg	5.0	10	300
Aroclor 1260	ug/kg	5.0	10	200
Aroclor 1262	ug/kg	5.0	10	--
Aroclor 1268	ug/kg	5.0	10	--

Notes:

1. -- = Not available or not applicable.
2. MDL = Method detection limit (MDL).
3. MRL = Method reporting limit (MRL).
4. JSCS = Screening levels from Portland Harbor Joint Source Control Strategy – Final (Table 3-1 Updated July 16, 2007). December 2005.



NOTE: Base map prepared from USGS 7.5-minute quadrangles as provided by Topozone. (1990)

0 2,000 4,000
Approximate Scale in Feet



Facility Location Map

Proposed Surface Soil Sampling
Swan Island Upland Facility Operable Unit 5
Portland, Oregon



Apex Companies, LLC
3015 SW First Avenue
Portland, Oregon 97201

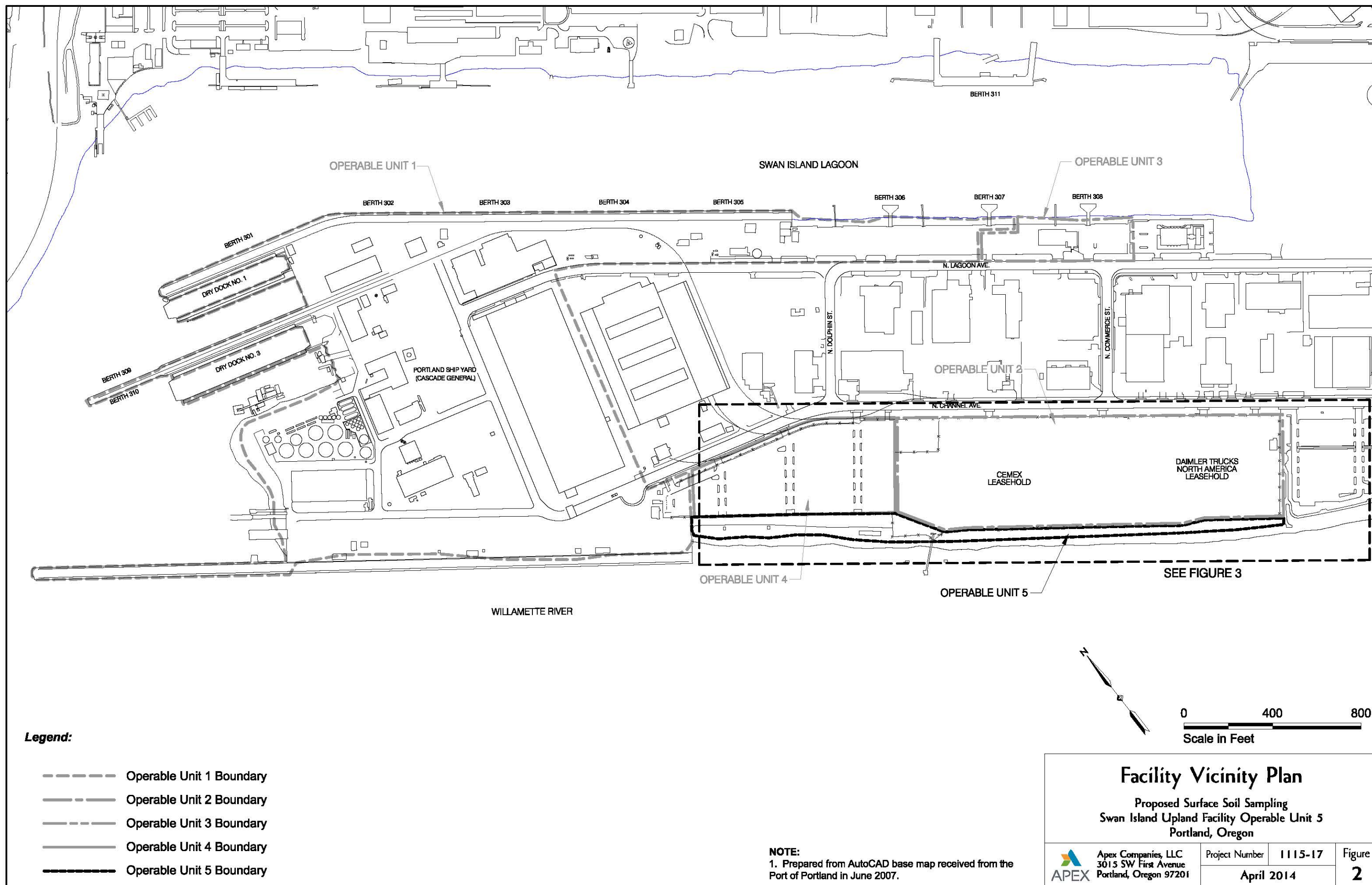
Project Number

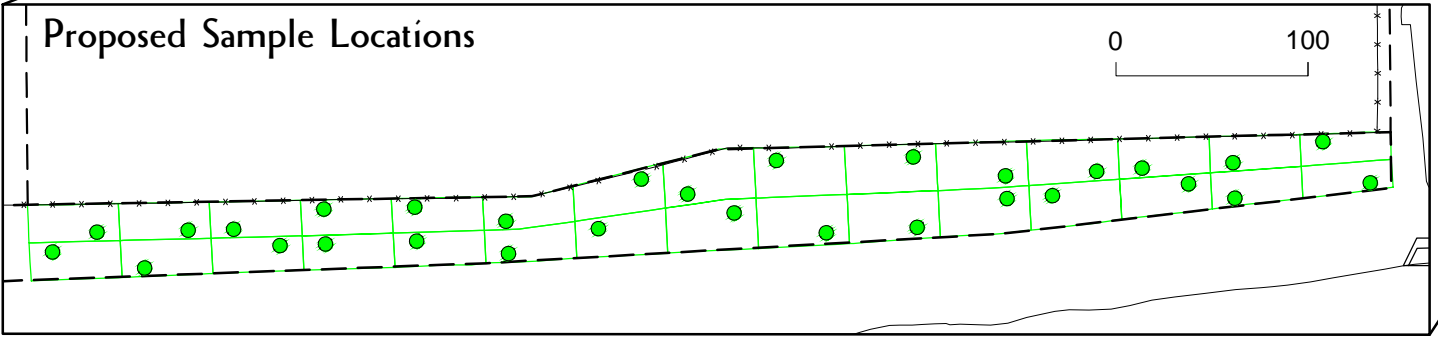
1115-17

April 2014

Figure

1





- Legend:**
- Proposed Incremental/Discrete Sample Location
 - WR-399 Outfall Location and Designation
 - Storm Water Pipe Location and Designation (Abandoned July 2006)
 - RB-8 Riverbank Soil Sampling Location (RB-8 through RB-15)
 - RB-4 Historical Composite Riverbank Sample Location (RB-1 through RB-7)

NOTES:
 1. Prepared from AutoCAD base map received from the Port of Portland in June 2007.
 2. Aerial photograph from 2012 - Google Imagery dated August 20, 2011.

0 200 400
Scale in Feet

Proposed Sampling Plan			
Proposed Surface Soil Sampling Swan Island Upland Facility Operable Unit 5 Portland, Oregon			
Apex Companies, LLC 3015 SW First Avenue Portland, Oregon 97201	Project Number	1115-17	Figure
	April 2014		3

Attachment A

Apex Labs Standard Operating Procedure – Incremental Sampling Methodology

APEX LABORATORIES, LLC

**STANDARD OPERATING PROCEDURE
APPROVAL SIGNATURE PAGE**

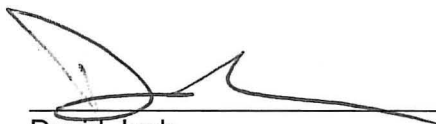
SOP Title: **Incremental Sampling Methodology (ISM)**

SOP Number: **GS-103 R0 ISM**

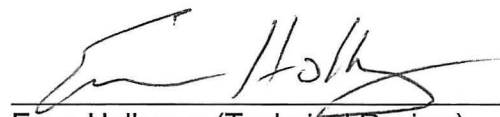
Effective Date: **June 3rd, 2011**

Approval Signatures:

Technical Manager:


David Jack 6/3/11
date

QA Manager:


Evan Holloway (Technical Review) 6-3-11
date

1 INTRODUCTION

This SOP describes the policies and procedures of Apex Laboratories concerning the preparation of soil samples received from Incremental Sampling Methodology (ISM) events. ISM is a sampling procedure that relies on a large number of samples (typically greater than 30) being collected in a certain area and combined into a single sample, rather than a smaller number of discrete samples that are analyzed individually. This procedure involves preparation of the combined sample and differs from normal lab compositing.

2 SCOPE AND APPLICATION

This procedure is typically applicable for analysis of metals and non-volatile organics. Preservation of samples for volatile organic analysis (VOA) is performed in the field. Compositing of preserved ISM VOA samples is not covered by this version of this SOP, which will be revised as necessary. See ITRC guidance for further information on VOA sampling and compositing.

ISM is a very project specific procedure, and should be driven by the client's Sampling Analysis Plan. Contact with the client is essential prior to beginning ISM processing, as the end use of the data may significantly change the procedure used to composite the samples. This SOP is intended as guidance for the steps common for most ISM samples, and is not intended to supersede client instructions as to how their samples should be handled. Modifications will be documented on the ISM request form (example, Appendix A).

3 SUMMARY OF METHOD

The entire volume of each sample is used in this preparation. The samples are air dried, then sieved through a #10 (2 mm mesh) sieve and the material that does not pass through is discarded. The material is either extracted and analyzed as is or further prepared for metals extraction.

4 SAFETY AND ENVIRONMENTAL

- 4.1 Personal protective equipment (P.P.E.) such as lab coats, nitrile gloves, and safety glasses must be worn while working with samples. Dust masks are optional, but recommended.
- 4.2 All secondary containers used to store samples or solutions beyond immediate use require proper labeling.
- 4.3 All waste, rinsate, expired solutions and/or solvents generated by this method should be handled in accordance with Apex's hazardous waste procedures. Care should be taken not to discharge any potentially hazardous or unknown substances into the drains or sinks.
- 4.4 Any step that creates dust, such as sieving or grinding, must be performed in a fume hood.

5 APPARATUS AND MATERIALS

- #10, #20 or other sieves
- Stainless steel bowls and spoons
- Ceramic mortar and pestle, Automated or Manual
- Dish and Puck Mill
- Aluminum baking sheets
- Heavy duty aluminum foil
- Butcher's paper
- Flat metal spatulas
- Sieve cleaning brushes
- Lab grade acetone or methanol

6 PREPARATION FOR PROCESSING

6.1 CLIENT CONTACT

An ISM coordinator will be designated for each project. This person will be the main client contact at Apex for the duration of the ISM event, and will supervise and review all steps of the process that occur at Apex and any portions of the processing that are subcontracted.

The ISM coordinator **MUST** contact the client regarding appropriate sample handling procedures and fill out an ISM Request Form. This should be done significantly prior to samples being received at the laboratory to allow for modifications of the method or apparatus as necessary.

The client's Sampling and Analysis Plan (SAP), however named, and DQOs must also be received by the laboratory prior to sample processing.

The ISM coordinator will also generate a project specific ISM Worksheet (example, Appendix B) to use as a template for the ISM process. This spreadsheet will act as a guide for sample login by designating the appropriate log in procedure and will outline the steps required by the client's SAP.

Effective communication between the lab, the samplers, and the project team is essential to a successful ISM project.

6.2 SAMPLE RECEIPT

6.2.1 ISM samples will be received either in multitudes of individual soil jars (at least 30) or in multiple bags containing samples pulled from at least 30 sites. These containers will generally not be logged in to Element as being associated with the sample work order or sample number. The sample referenced by Apex for all analyses will be created by this procedure. Log samples in for the Incremental Sampling Methodology test code, and create empty sample jars with labels in accordance with the ISM worksheet.

6.2.2 Once the ISM procedure is complete, the jars will be returned to sample receiving and

requested analysis can be added to the appropriate samples.

6.3 BLANK SAMPLE

6.3.1 A Blank sample consisting of Ottawa Sand will be processed through most steps of the ISM procedure along with the samples IF metals analysis is requested. It will be analyzed for metals only unless otherwise specified by the ISM worksheet. All references to a sample in the following steps will also include the Blank sample.

6.3.1.1 Due to volume restrictions, some steps of the process are not applicable to the blank. Note any steps not performed on the ISM worksheet. 1-D Japanese Slab Cake Subsampling is performed by default, 2-D Slab Cake is not applicable for the small volume used for the blank.

6.3.2 The Blank sample should be logged in as the last two samples on each work order where ISM will be performed. The first of the two Blanks will be processed as a sample by ISM. It will be provided to Sample Receiving in a 1 gallon plastic bag. The second will be analyzed as is in order to provide a baseline for metals analysis, and will be provided in a 4 oz jar.

6.3.3 Log in jars for the first of the two Blank samples according to the following table:

Jar A	Plastic Bag	Blank	<2mm	NA	No analysis
Jar B	4 oz jar	Metals analysis	Requested final grain size	> 15 g	Requested metals

6.4 EQUIPMENT CLEANING

6.4.1 All equipment and work spaces must be cleaned before and after each sample is processed in order to minimize the potential for cross contamination. The fume hood used for sieving and grinding must have its work surface and inside walls washed with soap and warm water and rinsed with acetone initially and between preparation of each composite batch of samples. All equipment should be washed with warm water and soap before and in between each sample batch, followed by a rinse with acetone.

6.4.2 Trays used for air drying, subsampling, etc. may be lined with new aluminum foil or butcher's paper prior to use instead of the above cleaning procedure.

6.4.3 All references to cleaned equipment indicate that one of these procedures should be followed before use.

7 SAMPLE PROCESSING

In order to reduce potential sources of error, this procedure processes the entire sample received

at the laboratory through as many steps as possible. Unless otherwise specified, references to sample in this document refer to the total amount of sample received, or what is still defined as sample after prior processing steps. See the Quality Control section for a further discussion on sources of error and Data Quality Objectives (DQOs).

Each ISM sample will be different. The following steps are potential parts of any ISM processing, but may not be used for all samples. As such, the processing for each ISM sample will be driven by the SAP and the steps below should not be considered sequential requirements for all ISM projects. Refer to the SAP and the ISM worksheet for which steps are necessary for each sample. Steps not included in this SOP may be necessary. Details of these steps should be included in the ISM worksheet or other documentation.

7.1 SAMPLE IDENTIFICATION

ISM samples may include material that is not considered part of the analytical sample. Vegetation, oversized material, and decantable water are examples of material that may be requested to be removed before sample processing begins. The SAP should include detailed instructions as to what defines the analytical sample, and what to do with materials that are removed. This may include documenting their removal photographically, and potentially by weight.

7.2 PERCENT MOISTURE DETERMINATION

If as received percent moisture determination is requested on samples, it must be performed before samples are air dried. Samples will be homogenized as best as possible with field moist samples, and a subsample aliquot taken as using the 2-D Japanese Slab Cake method. This may be done with or without wet sieving.

This result will be reported as the percent moisture. Dry weight analysis and correction will be performed on the prepared samples, but this result does not reflect the percent moisture of the sample as received.

7.3 SAMPLE SPLITTING/MASS REDUCTION

Splitting an ISM sample may be requested prior to other processing in order to have two separate sample processing paths for two different types of analysis, for sample mass reduction, or other reasons. This is not recommended due to potential increases in uncertainty of the data. Duplicate field samples are the preferred method for separate processing steps.

7.3.1 Three simple sample splitting techniques are available for use at Apex:

7.3.1.1 Alternate Shoveling divides the sample into two subsamples by placing alternate subsample scoops of the original sample into two separate sample containers.

7.3.1.2 Fractional Shoveling is similar to alternate shoveling except the sample is divided into three or more subsamples.

7.3.1.3 Cone and Quartering splits the sample into two subsamples by pouring the sample into a large cone, flattening the top and dividing into four sections. Opposite sections of the sample are then combined to form the two subsamples. This requires a flowable sample, and should be performed after samples are air dried and disaggregated. Therefore, this is only an option if both sample splits can be air dried.

7.4 SAMPLE CONDITIONING

Sample conditioning is usually necessary before homogenization or particle size reduction steps, in order to produce a flowable sample. Some sample conditioning steps may not be appropriate for some Chemicals of Concern (COCs), such as low boiling point SVOCs and Mercury. (See ITRC Table 6.1.) The SAP should address acceptable sample conditioning steps and how to process samples if conditioning is not acceptable.

Air drying at room temperature is the default sample conditioning step used by Apex if particle size reduction steps such as sieving are required. Other conditioning steps include drying at elevated temperature, freeze drying, and water addition. If these methods are requested, their procedure should be carefully specified in the SAP.

7.4.1 AIR DRY

7.4.1.1 Air dry the entire volume of all the sample containers by emptying them out on flat aluminum bakers sheets lined with heavy duty aluminum foil or butcher's paper and spread out to a depth of < 1 inch.

NOTE: Aluminum may not be an appropriate choice for samples where aluminum, chromium, or other compounds that may react with aluminum, are COCs. Paper or plastic maybe better choices in these cases. However, plastic must be avoided if phthalates or placsticizers are COCs, and paper cannot be used if organic carbon or other organics that may sorb to paper are COCs.

7.4.1.2 Place trays in bakery rack and allow to dry at ambient temperature in a low traffic area with sufficient air flow to carry away evaporated moisture, such as in or near a fume hood. 1-2 days are normally needed. Turning samples may be necessary to aid the drying process for wet samples, and layers of clay should be broken up in a mortar and pestle halfway through the drying process to avoid formation of bricks that are difficult to break apart after they are fully dried.

7.4.1.3 Samples should not be allowed to dry for more than three days, due to potential loss of more volatile analytes.

7.4.1.4 Record the air drying start and end times on the ISM worksheet.

7.4.1.5 After samples are dry, remove any visible sticks, rocks, vegetation, or other non-soil materials.

NOTE: If samples will be air dried, they do not need to be stored in the refrigerator. However, they most likely will be for ease of sample control. Ask the Sample Control department if questions arise about appropriate sample storage locations.

7.5 PARTICLE SIZE REDUCTION

For many projects, particle size reduction will be required in order to reduce the uncertainty associated with the data. Most samples will require that the particle size is less than 2 mm before analysis. This will ensure that a 10 to 30 gram aliquot will be enough sample volume to meet DQOs. For analyses that cannot use at least 10 grams of sample, (metals, cyanide, and other wet chem tests) grain size of less than 0.25mm must be achieved. Specific projects may require even finer grain sizes for these analyses.

If the ISM worksheet specifies that the sample will be processed to reduce particle size, there are many techniques that may be used. Automated mortar and pestle or dish and puck mill are two that are available to Apex. Depending on the COCs, these may not be appropriate, and SAP should specify which technique to use.

If a particle size reduction step is required, the entire sample should be ground so that it can pass through the sieve corresponding to the final grain size requested by the ISM worksheet. If multiple analyses are to be performed, this may require multiple samples to be taken in the field, or the sample to be split prior to processing.

7.5.1 SAMPLE SIEVING

7.5.1.1 Soil clumps should be broken up to allow them to pass through the sieve, and anything remaining in the sieve (stones, metal, glass) should be discarded and noted. Clay, wet, and/or rocky samples pose significant difficulties during this process. Breaking up dried clumps of dirt/clay and separating them from the material to be removed may be facilitated by grinding, pounding, tumbling or shaking samples by any available means. Record procedure used on ISM worksheet.

7.5.1.1.1 A sieve stack consisting of a lid, #4 and #10 sieves and a sieve pan may be loaded with sample and placed in to a sieve shaker for 5 to 10 minutes to breakup clumps without changing particle sizes.

7.5.1.1.2 A blender or coffee grinder may be used to disaggregate samples, but keep blending times low to reduce wear on blade, contamination of samples with blade material, and loss of analyte due to sample heating.

7.5.1.1.3 A mortar and pestle may be used, though this method can cause more particle size reduction than other methods.

7.5.2 MILLING/GRINDING

This step is often done on the sample that has passed through the #10 sieve. (Everything larger than 2mm is not defined as sample.)

7.5.2.1 Automated Mortar and Pestle: Using a cleaned mortar and pestle, grind the entire sample until it is fine enough to pass through the required sieve, as noted on the ISM

worksheet. See instrument manual or Apex operating procedure for details.

NOTE: This can also be done manually, which is a very laborious process and should only be done for small samples with few particles greater than the required size.

7.5.2.2 Dish and Puck Mill: This may be appropriate for some projects where metals are not COCs. See instrument manual or Apex operating procedure for details.

7.5.3 Enter details of the operation, operator initials and date on the ISM worksheet.

7.6 HOMOGENIZATION

The sample mixing step specified here assumes that the sample has been sieved so that all particles are less than 2mm. If this is not the case, simply stirring the sample will be more likely to increase sample homogeneity than decrease it, due to particle size separation within the bowl. Tumbling the sample in a container with sufficient headspace to allow free movement, or placing the entire sample into a blender or mill are better options in the case of un-sieved samples.

7.6.0.1 Place the entire sample (minus any portions removed during the Air drying and Sieving steps, if performed) into a stainless steel bowl. Stir the sieved sample well (approximately 3 minutes) to homogenize.

7.6.0.2 If it is necessary to complete the compositing procedure at a later time, place the entire homogenized sample into the 1 gallon re-closeable plastic bag labeled A for storage.

7.6.1 Enter operator initials and date on the ISM worksheet.

7.7 SUBSAMPLING

There are many methods available for subsampling, some of which produce less error than others. Apex has available two simple incremental sampling methods. If other methods are required, Apex will procure the appropriate technology or subcontract this portion of the process.

If subsampling for an analytical aliquot, pay close attention to the ISM worksheet. The aliquots taken must be very close to the mass requirements, because the entire aliquot subsampled must be used for analysis.

If specified by the ISM worksheet, repeat this process as needed to provide sample volume for process duplicate or triplicate analyses.

7.7.1 1-D JAPANESE SLAB CAKE

7.7.1.1 Pour the entire sample into a line, using 20 or more passes along the line to distribute the sample. For samples where small analytical masses are required (e.g. metals, cyanide) a long thin line should be created.

7.7.1.2 Using a square scoop, cut across the line to create an aliquot. Combine as many of these aliquots as needed to create the analytical sample or mass reduction required. Repeat until all analytical aliquots have been created.

7.7.1.3 Place the aliquots into their respective containers, according to the ISM worksheet.

7.7.1.4 Place the remainder of the sample into the 1 gallon re-closeable plastic bag labeled A for storage.

7.7.2 2-D JAPANESE SLAB CAKE

7.7.2.1 Pour the entire sample into a cleaned aluminum tray and spread evenly. Use a pre-formed grid with 30 sections to divide the sample. Pull an equally sized aliquot of sample from each section of the grid and combine into the appropriate container for analysis. Be sure to scrape along the bottom of the tray in order to include a representative portion of all grain sizes present in the sample.

7.7.2.2 Pull an aliquot of sample from each section of the grid to ensure that the final sample size is close to the mass requested for analyses, typically 10-30 grams. Place the aliquots into their respective containers, according to the ISM worksheet.

7.7.2.3 Place the remainder of the sample into the 1 gallon re-closeable plastic bag labeled A for storage.

7.7.3 When subsampling is complete, roll the jar(s) for 1 minute to homogenize the sample. Initial and date the ISM worksheet.

7.8 DOCUMENTATION:

7.8.1 Create a batch in Element for the ISM test code, add the samples processed as a batch, and print out the bench sheet. Set sample status to Needs Review, attach the completed ISM worksheet and submit for review and scanning.

7.8.2 Return jars to Sample Receiving for completion of log in.

7.9 LOG IN

7.9.1 After samples are returned from ISM processing, analysis test codes can be added to the samples.

7.9.2 Be sure to add comments indicating the use for each jar in accordance with the ISM worksheet. Because one jar will be created per analysis, duplicate, and MS/MSD, there will be a large number of containers for some samples. The container comments should match the ISM worksheet, and the work order should be reviewed carefully by the person coordinating the ISM project.

7.10 ANALYSIS

Each aliquot for analysis has been pulled during sample processing and placed into a separate container. Use the ISM worksheet and the analysis comments to find which container is designated for your analysis. Be sure to use the entire amount of the aliquot provided, and rinse the container into the extraction vessel. Check the sample comments for sample specific instructions (e.g. MS/MSD, etc).

8 QUALITY CONTROL

8.1 FUNDAMENTAL ERROR

The steps in this procedure are designed to ensure that the fundamental error (FE) associated with the sample is below 15% in the final aliquot used for extraction and analysis. This FE measure has been determined to be the primary lab DQO.

Fundamental Error is calculated using the following equation:

$$FE = \text{Square Root}((20 * d^3)/m)$$

Where:

20 = sampling constant

d = maximum sample grain size (cm)

m = sample mass used for extraction and analysis (g)

For samples taken from the - #10 sieve fraction, d = 0.2, m = 10 and FE = 12.6%
d = 0.2, m = 20 and FE = 8.9%

For samples taken from the milled fraction, d = 0.0850, m = 1 and FE = 11.1 or
d = 0.0250, m = 1 and FE = 1.8%

8.2 CONVENTIONS

8.2.1 Samples will be reported on a dry weight basis. The reported dry weight result will reflect the moisture left in the sample after air drying.

8.3 QUALITY CONTROL SAMPLES

8.3.1 Blank: A blank using Ottawa sand is processed and analyzed along with samples tested for metals to verify that no contamination is being added by processing the samples. This will be done as requested for other classes of COCs.

8.3.1.1 The Ottawa sand will have to be tested before and after processing to compare levels of metals present, as no known clean matrices for metals exist.

8.3.2 Process Replicates: Whether process replicates will be analyzed should be determined by the client on a project basis. They may request that one or two replicates be performed per

project, per batch, or per sample.

8.3.2.1 Aliquots may be pulled and designated to be analyzed as batch duplicates in the same manner as sample aliquots. This should be specified on the ISM worksheet, as a separate container will have to be created for them.

8.3.3 Matrix Spikes: Apex will not evaluate spike samples through the entire ISM process unless requested. If required to do so by a client, the client should specify or provide a standard reference material suitable for ISM processing.

9 REFERENCES

- 9.1 Hawai'i Department of Health *Technical Guidance Manual for the Implementation of the Hawai'i State Contingency Plan*, Section 4, November 12, 2008.
- 9.2 Alaska Department of Environmental Conservation Division of Spill Prevention and Response Contaminated Sites Program *Draft Guidance on Multi-Increment Soil Sampling*, March 2009.
- 9.3 EPA Method 8330B Appendix A Revision 2 October 2006.
- 9.4 Interstate Technology Regulatory Council *Technical and Regulatory Guidance: Incremental Sampling Methodology*, March 2011 (Draft)

Appendix A – Example ISM Request Form

Client:	_____	Notes:
Project:	_____	
Client Contact:	_____	
# of Decision Units:	_____	
# of Increments / Unit:	_____	

Analysis: Note any that require subcontracting or small sample size (e.g. Metals)

Which ISM guidance document is being used for this project?

Alaska

Hawaii

EPA 8330A Appendix A

ITRC Draft ISM Guidance

When will the Sampling and Analysis Plan be completed?

A copy must be provided to Apex before the project begins.

Project Specific Data Quality Objectives and procedures.

Apex follows the ITRC Draft guidance where possible. The following categories are procedural steps that are likely to have project specific goals. Our standard procedure is listed under the Apex heading, followed by specific requirements from the guidance documents. Each sampling event is unique, and modifications from our default procedure are expected. These differences should be noted.

Sample Storage:

Apex: Store refrigerated until air drying, room temperature thereafter.

Client request?

Air Drying:

Apex/ITRC: Air dry samples to help with sieving and grinding. Consider potential effects on volatile Contaminates of Concern (COCs) such as SVOCs and Mercury.

AK: Air dry only if necessary to sieve to < 2mm. May not be appropriate for Pesticides and PAHs.

HI: Air dry for all non-volatile analytes.

Client request?

Dry Weight:

Apex/ITRC: Samples are air dried, sieved, and then subsampled. That subsample is tested for most analysis and for dry weight. Results are reported on a dry weight basis, corrected to the air dried sample. If field percent moisture is requested, then a separate aliquot must be made prior to air drying.

HI: Air dried = dry weight, no further correction needed.

Client request?

Appendix A – Example ISM Request Form

Fundamental Error / Sample size:

Apex: Our goal is to have less than 15% Fundamental Error at all steps. Our particle size and sample mass requirements are chosen to meet this goal for each analysis.
We use at least 10 grams and generally ~20 for most tests, with a particle size smaller than 2 mm.
We try to use at least 1 grams for Metals and other limited volume tests, with a particle size less than 250 μm .

AK: Requires at least 30 grams of sample, particle size smaller than 2 mm.

HI: At least 10 grams for most tests, particle size smaller than 2 mm.
At least 1 gram for Metals and other limited volume tests, particle size less than 250 μm .

ITRC: Somewhat contradictory. Generally, 10 grams for <2 mm fraction, 2 grams for < 0.25 mm.

Client request?

Project Specific Fundamental Error (FE) goal?

Laboratory Replicate Samples:

Apex: Per client SAP.

ITRC: Field and lab triplicates are recommended for most projects.

Client request?

Blank:

Apex/ITRC: We have a blank sand matrix go through all steps of the analysis to ensure that metals are not added by the ISM process. Other analysis can be performed on the blank at additional cost. Matrix spikes are performed on a batch basis, per analysis.

Matrix Spikes:

ITRC: Suggests that processing standard reference materials may be appropriate for some projects and COCs.

Notes:

Appendix B – ISM Worksheet

Batch _____

Sample Log in

Each sample created by the ISM procedure will be logged in with the containers and comments specified below. If samples will be treated differently, multiple sections will need to be created.

Sample IDs:

	Container	Use/Analysis	Particle size	Weight Needed	Comments
Jar A	Plastic Bag	Composite	<2mm	NA	No analysis
Jar B	4 oz jar				
Jar C					
Jar D					
Jar E					
Jar F					
Jar G					

Air Dry

Sample ID	Analyst	# of Containers to Composite	Air Dry Start Time	Air Dry End Time	Comments (Note sticks, rocks, etc removed.)

#10 Sieve

Sample ID	Date	Analyst	Homogenized?	Comments

Appendix B – ISM Worksheet

Splitting or Subsampling

This section may be needed multiple times for each sample. Modify work sheet to include this section for each step.

Method Used: 1-D Japanese Slabcake 2-D Japanese Slabcake Alternate Shoveling Fractional Shoveling Cone and Quarter Other:

Sample ID	Date	Analyst	Replicates?*	Weight Obtained**	Homogenized?	Comments

*Indicate use for Replicates (Dry Weight, Duplicate analysis, etc)

**Total weight minus tare. (8 oz jar tare weight is 215g, 4 oz jar tare weight is 130g)

Grinding

This section may be needed for only a portion of each sample. Ensure that the proper container is noted.

Method Used: Automated Mortar and Pestle Manual Mortar and Pestle Dish and Puck Mill

Sample ID	Jar	Date	Analyst	Sieve size	Homogenized?

Sieve Size Chart

#10	2 mm
#20	850 µm
#40	425 µm
#60	250 µm
#100	150 µm
#140	106 µm
#200	75 µm

Comments:
